Assessing polymer powder flow for the application of laser sintering

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Abstract

A need exists for techniques to assess flow properties of powders intended for laser sintering (LS). Although several powder flow measurement techniques are available, the flowability of a powder is strongly dependent on the nature of the applied flow field and none of the currently availably techniques adopt the flow field of LS. Therefore, this paper proposes a new technique, which mimics the flow in an LS machine, allowing a more appropriate powder flow evaluation for this particular process. The set-up provides qualitative assessment of powder layer smoothness, as well as a quantitative determination of the packing density of the deposited layer. Measurements on PA12, spherical PS and PMMA, and cryogenically milled TPU powders demonstrate the set-up's capability to evaluate powder flow with regard to LS.

Keywords: Laser sintering, Powder flow, Powder spreader, Polymers, Powder characterisation

1 Introduction

Due to the complex nature of powders, it is common that different flow measurement techniques, with hence different stress states, yield dissimilar sample classifications [**1**,**2**]. Hence, to obtain informative results for a certain application, the chosen technique requires a flow field as similar as possible to the intended application. Here, the application of interest is laser sintering (LS).

LS is a form of Additive Manufacturing, a set of techniques in which parts are built layer-by-layer [**3**,**4**]. As a base material, LS uses powders with typical particle diameters of about 50 µm [**3**,**5**,**6**]. The LS machine spreads the powder into thin layers of around 100 µm in thickness. In each layer, a laser sinters the part's cross-section according to a 3D model. The cycle of spreading and sintering repeats until the part is finished [**3**,**6**]. It is crucial that the deposited layers are smooth, show no surface defects and preferably have a high packing density in order to reduce the part's porosity [**3**].

On an industrial scale, the layer quality is often determined by trial-and-error. For the determination of the packing density, which is affected by the powder flow quality, industrial LS machines sinter closed hollow cubes. A weight determination of the enclosed, unsintered powder provides the packing density [**7**]. This technique, however, requires a large amount of powder and the investment in a fully operational LS machine. To our knowledge, no specific lab-scale testing methods exist to assess the powder flow quality in the sinter process, as was also formulated by Schmidt et al. in earlier studies [**8**]. The aim of this research is to introduce a useful technique to determine powder flow quality for the laser sintering process.

2 Methods and materials

2.1 Powder spreader

In this work, a new lab-scale powder spreader device is introduced to measure the powder flow quality, particularly for the LS process. The set-up, illustrated in Figure 1, mimics the layer deposition of a commercial LS machine and assesses the surface quality and packing density. A powder sample is loaded in front of the spreading blade, after which this blade deposits the powder into a thin layer on the measurement plate. The thickness of this layer is imposed by the difference in height between the spreading blade and the measurement plate. Both heights can be adjusted separately, allowing the study of different layer thicknesses, as well as multi-layers. The latter is crucial, as it enables the study of powder-on-powder deposition, which is the type of flow encountered in LS. The measurement plate, measuring 14 cm by 17 cm, rests on a balance. As a result, the balance provides a measurement of the layer weight and, as the layer dimensions are known, the layer density (ρ_{layer}). Dimensions are optimised to provide a sensitive measurement with a minimal amount of sample. Experiments suggest that a scale precision of 0.01 g is adequate to obtain significant measurements.

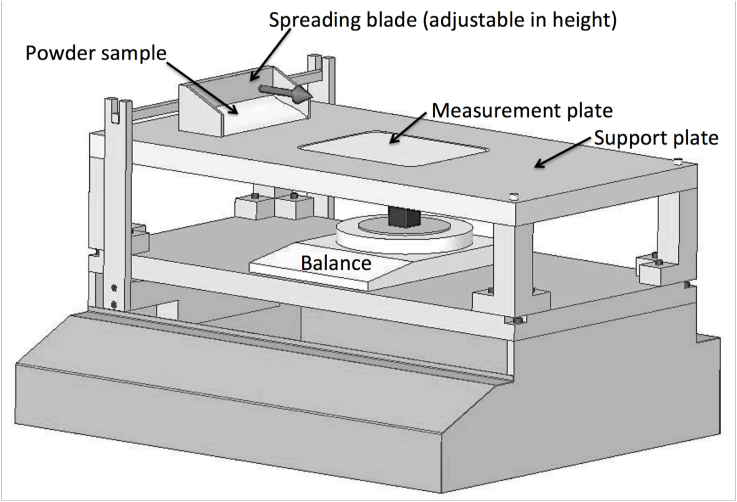

Figure 1: schematic illustration of the powder spreader set-up.

The set-up is created by the modification of a commercial Elcometer 4340 Motorised Film Applicator. The main adjustments include a support plate for carrying the spreading blade and the aforementioned measurement plate, which rests on the balance. An Elcometer 3580 Casting Knife Film serves as the spreading blade. This applicator consists of a spreading blade, fixed between two blade holders, which rest on the support plate. The spreading blade can be raised or lowered by a micrometer, calibrated at 0 µm where the blade touches the support plate and with a precision of 10 µm, thereby creating a gap of a known height. The set-up however allows the use of other application mechanisms, such as the applicators used in actual LS machines.

2.2 Powder flowability

Both powder flowability and the densest geometrical packing affect the layer density (ρ_{layer}). The better the powder flows, the smaller the particle interactions are, resulting in fewer voids and thus a larger density. ρ_{layer} is directly relevant to the LS process, as a high layer density reduces part porosity, and improves the final part accuracy [**9**,**10**,**11**]. To exclude the contribution of the material density and thus allow a quantitative comparison of powders of different polymers, a dimensionless packing density, ρ_{p}, is defined as the ratio of ρ_{layer} and the material density of the polymer (ρ_{m}).

$$\rho_{\mathrm{p}} = \frac{\rho_{\mathrm{layer}}}{\rho_{\mathrm{m}}} \tag{1}$$

The particle geometry and size distribution create a lower limit for the amount of voids. To exclude also these geometrical limitations and look solely at powder flowability, it is useful to define the maximal packing density. Tapping a container of powder approximates the maximal possible packing. During the taps, the particles temporarily lose contact and improve their packing due to reduced friction. This dense packing is referred to as the tapped density (ρ_{tap}) [**12**]. The ratio of ρ_{tap} to ρ_{m} thus provides the upper limit for the packing density, limited not by flow, but by geometrical restrictions.

$$\rho_{\mathrm{p,max}} = \frac{\rho_{\mathrm{tap}}}{\rho_{\mathrm{m}}} \geq \frac{\rho_{\mathrm{layer}}}{\rho_{\mathrm{m}}} = \rho_{\mathrm{p}} \tag{2}$$

The ratio of the packing density to its upper limit now provides an index, the packing ratio (PR), which excludes the geometrical limitations as well as the material density and thus only looks at powder flowability. The same ratio is found by dividing layer density directly by the tapped density.

$$\mathrm{PR} = \frac{\rho_{\mathrm{p}}}{\rho_{\mathrm{p,max}}} = \frac{\rho_{\mathrm{layer}}}{\rho_{\mathrm{tap}}} \tag{3}$$

Notice the analogy with the Hausner Ratio, a widely used indicator for powder flow $\frac{1}{\mathrm{HR}} = \frac{\rho_{\mathrm{bulk}}}{\rho_{\mathrm{tap}}}$, where ρ_{bulk} is the bulk density of the powder. The indices introduced in equations 1 and 3 are hence not real new indices but rather an adaptation of existing indices to LS. However, the HR utilises the bulk density of a freely poured powder [**13**]. The flow field thus strongly differs from the forced spreading in LS, which makes the HR less relevant for this application.

Summarised, ρ_{p} provides a directly useful index for the packing quality of a powder layer in LS. Higher values are preferable, as these lead to denser sintered parts. PR, on the other hand, provides an index that solely evaluates powder flow in LS. As both the geometrical packing and powder flow are relevant, this study reports on both indices.

2.3 Methodology

An experimental protocol is designed to compare the flow behaviour of different powders. Before the actual test, the measurement plate is positioned 1 mm below the upper surface of the support plate. The spreading blade rests on the support plate and the powder is gently poured in front of the blade. The blade is then pushed across the plate at a selected speed, depositing a powder layer on the measurement plate. The first layer serves as a base layer on which subsequent layers are deposited to create conditions similar to those in an LS machine. Moreover, this ensures that any subsequent layers are deposited on a perfectly levelled area.

For the following layers, the blade is raised 100 µm each time, allowing the deposition of a 100 µm layer. An amount of powder, roughly twice the amount needed to form a 100 µm layer, is poured in front of the spreading blade. After the deposition, the balance measures the added weight, which serves as a first data point. This process is repeated, and subsequent layers are spread to gather additional data points. The results are obtained by taking the average over twenty layers. The layer quality is observed visually. The presence of defects is noted, as well as a qualitative indication for the surface roughness.

The spreading velocity in this study is kept at 3 cm/s, however, the set-up allows for the use of different spreaders and spreader velocities enabling the user to optimise the spreading mechanism for each specific powder and to mimic the flow in an LS machine as closely as possible. Preliminary tests note little difference for a velocity variation between 1 cm/s and 14 cm/s, which are the limits of the current set-up.

ρ_{tap} is obtained by filling a 20 ml container with the powder and manually tapping it until no further contraction is visible. This final volume is measured and divided by the sample weight [**14**]. The manual tapping procedure poses some accuracy limitations. Preliminary tests with an automated tapping protocol and a container volume of 100 ml, show a density difference of less then 3 % for the PA powders. The same powder ranking was obtained.

2.4 Materials

The powder spreader is tested on various sets of powders, covering a wide range of distinct morphologies and particle sizes. Figure 2 shows microscopy pictures of all investigated powders. Table 1 provides the median diameters, obtained from laser diffraction experiments, as well as the span, defined as $\frac{D_{90}-D_{10}}{D_{50}}$.

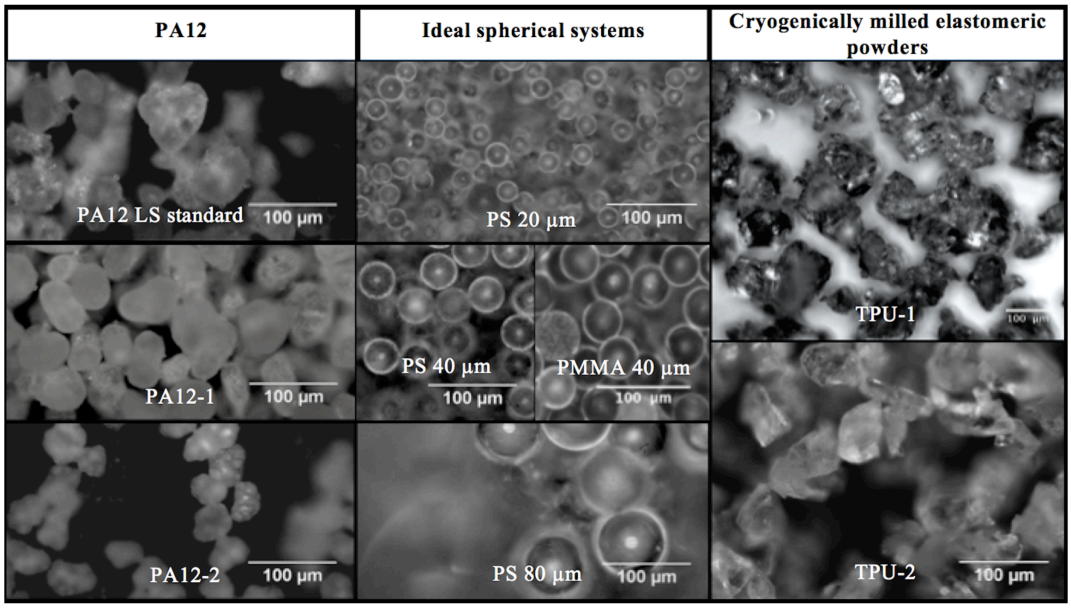

Figure 2: microscopy pictures of the investigated powders.

The first set of powders consists of three different polyamide 12 (PA12) powders. As this material currently dominates the LS market [**3**], it will be used as a benchmark for this study. One standard PA12 grade for laser sintering is provided, with a mass-median-diameter of 59 µm. Two other PA12 powders, further referred to as PA12-1 and PA12-2, are included. PA12-1 has smoother particle edges than the LS grade, whereas PA12-2 has a morphology comparable to the LS grade, but has a smaller average particle size, around 42 µm.

Another set consists of perfectly spherical particles. Polystyrene (PS) spheres with diameters of 20 µm, 40 µm and 80 µm are tested. To provide additional insights, also polymethylmethacrylate (PMMA) spheres of 40 µm are included. The PS and PMMA powders are not directly relevant for LS, but as they consist of rather monodisperse spheres, they form an ideal reference system.

A final set consists of two cryogenically milled TPU powders, further referred to as TPU-1 and TPU-2. As both powders were milled, they consist of very irregular particle shapes and sizes, but TPU-1 has a slightly larger average particle size than TPU-2.

3 Results and discussion

Figure 3 illustrates representative layer surfaces for the aforementioned powder sets. The PA12 LS standard exhibits a slightly rough, though homogenous surface. The other PA12 powders behave similarly, and all the PA12 powders deposit into defect-free layers. Also the spherical powders form defect-free layers. Moreover, these layers are much smoother than those of the PA12 powders. The cryogenically milled samples form layers of poor quality. TPU-1 is unable to form complete layers under the given conditions. TPU-2 deposits into layers, but shows severe defects in most cases.

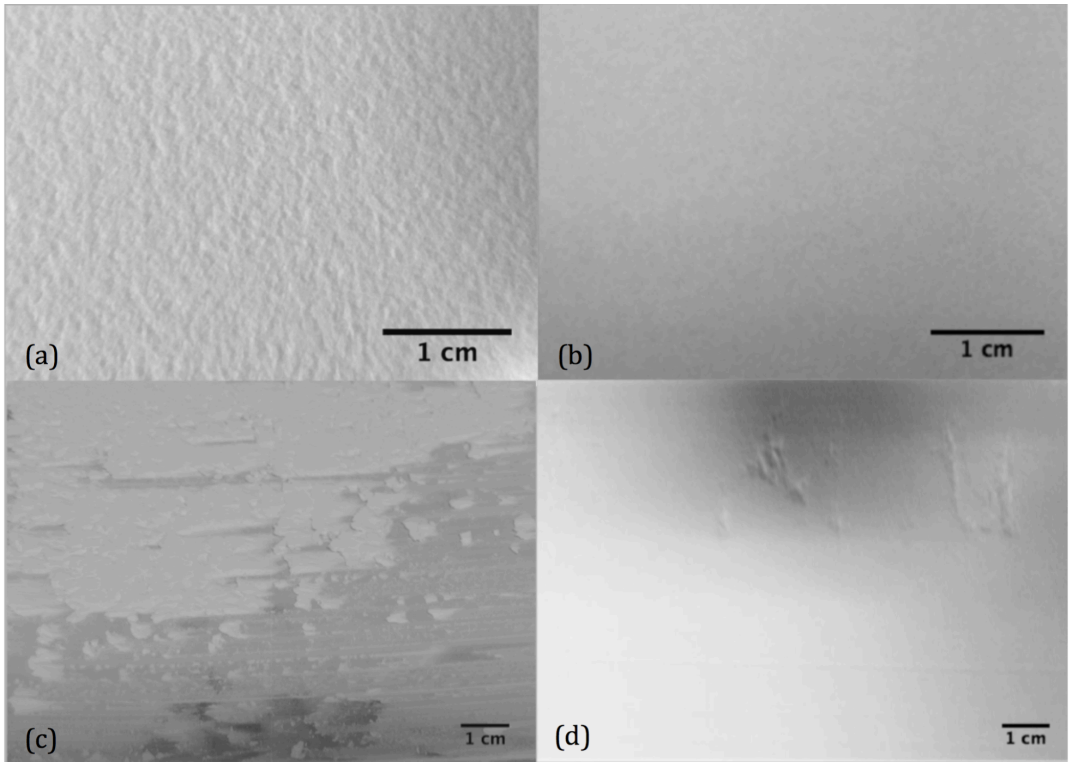

Figure 3: pictures of observed layer quality (a) PA12 LS standard (b) PS 40 µm; PS 20 µm and PMMA 40 µm powders showed visually identical surface qualities (c) TPU-1 (d) TPU-2

Table 1 provides the results for the packing densities. Again, the spherical powders show optimal behaviour, with PR well above 90 % for all samples. The 80 µm PS spheres, however, are too large to closely fill a gap of 100 µm, resulting in alternating high and low deposited layer masses, explaining the high standard deviation (18 %). When this powder is tested with a layer thickness of 240 µm, all other parameters kept identical, a much smaller variation is observed, proving this point.

Table 1: summary of the test results for all examined powders.

Parameter	**PA12 powders**			**Monodisperse spheres**				**Cryogenically milled**	
	PA12 LS standard	**PA12-1**	**PA12- 2**	**PS 20 µm**	**PS 40 µm**	**PS 80 µm**	**PMMA 40 µm**	**TPU-1**	**TPU-2**
D_{50} [μm]	59	55	42	24	44	83	42	69	70
Span [−]	0.84	0.74	0.58	0.56	0.51	0.46	0.31	1.43	1.44
ρ_{tap} $\left[\frac{kg}{m^3}\right]$	520	543	486	632	639	643	725	588	644
ρ_p [−]	44 %	47 %	40 %	57 %	59 %	58 %	60 %	No usable layer	53 %
PR [−]	88 %	90 %	85 %	93 %	97 %	94 %	98 %	/	87 %
σ_{PR} [−]	4.8 %	3.3 %	3.5 %	7.0 %	4.8 %	18 %	3.4 %	/	12 %
Layer quality	Slight roughness	Slight roughness	Slight roughness	Smooth	Smooth	Smooth	Smooth	Incomplete layer	Small defects

The PA12 samples show significantly lower values, 85 % - 90 %, which is not surprising taking into account the roughness of the deposited powder layers. PA12-1 and PA12-2 respectively show denser and less dense packing than the LS standard. However, the difference in tapped density indicates that this result is not only due to a different flowability, but also due to geometrical packing limitations. These affect the maximal density attainable, no matter the flow quality. This is also seen in a larger relative difference in ρ_p, 7 % difference on a value in the order of 45 %, than for PR, 5 % difference on a value in the order of 85 %, illustrating the benefit of reporting both indices.

Finally, TPU-2 shows a relatively high packing ratio, 87 %, even though the particles have very irregular shapes. The elasticity of the polymer may form an explanation, as TPU-2 proves to be very compressible during tests. When the spreading blade moves over the bed, it pushes the particles down, slightly compressing them. After the blade has passed, the powder relaxes again, leaving a powder layer with a slightly larger thickness than intended. Hence, the calculated packing density of elastic powders is not fully accurate. This compression and thickness uncertainty also results in a large standard deviation, even though it is still close to realistic LS conditions. If a more accurate packing density is desired, a measuring system for the actual layer thickness should be added.

A remark on these results is that they are obtained by using a non-optimised spreading blade. Preliminary tests with an in-house built spreader, which more closely resembles a recoating blade used in actual LS machines, showed that this spreader was able to form relatively homogenous layers of the TPU-1, although these layers were still not defect-free. Goodridge et al. also illustrate that for less flowable powders, the use of a counter-rotating roller is more forgiving than a recoating blade. [**3**].

The experiments demonstrate the usefulness of the powder spreader. From a practical point of view, the operation and understanding of the set-up is fairly simple, as well as the interpretation of the results. Conclusions on the flowability of the examined powders can be drawn without the need for a deep understanding of flow mechanics nor the use of advanced software. This makes the technique very accessible for researchers of various backgrounds. The amount of required sample is limited and depends on the number of layers to be formed. In order to get multiple data points and to minimise the boundary effect of the metal measurement plate, it is advised to place multiple layers. A minimal sample volume of 100 ml is advisable.

Unfortunately the current set-up still shows some shortcomings. This study does not include environmental control. However, the powder bed in a typical LS application is kept at a temperature close to the sintering temperature to minimise thermal gradients and part warpage [**3**,**13**]. Temperature is expected to have considerable influence on the flow properties, as does humidity. The addition of both temperature and humidity control is planned in future studies.

4 Conclusion

Even though many set-ups for the assessment of powder flow exist, none of them provide the direct link to laser sintering. The developed powder spreader is able to provide a direct assessment of powder flow in an LS machine. It mimics the flow on a small scale, assessing both the layer quality and the resulting packing density. Tests on three distinct sets of powders, namely PA12 powders, monodisperse spheres and cryogenically milled elastomers, prove the potential of this technique and stimulate its further development. The results show that the PA12 LS standard formed layers of a sufficient quality for LS, as to be expected by its dominance in the LS market. The other PA12 powders showed similar results, but some differences in packing density were observed. The monodisperse spheres acted as a reference, providing layers of a high smoothness and packing density. Finally, the cryogenically milled elastomeric powders showed clear problems in spreading, but illustrated how an optimisation of the spreading mechanism may overcome flow problems to a certain extent. Future studies will also encompass the addition of both temperature and humidity control to the current set-up.

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